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A study of base exchange on clays by electrodialysis

Collier, James Richard

Troy, New York; Rensselaer Polytechnic Institute

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A STUDY OF BASE EXCHANGE ON CLAYS
BY ELECTRODIALYSIS

JAMES R. COLLIER

Thesis
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A STUDY OF BASE EXCHANGE ON CLAYS
BY ELECTRODIALYSIS

James R. Collier
Ensign, CEC, USN

Presented as partial fulfillment of
the requirements for the degree of
Master of Civil Engineering
Rensselaer Polytechnic Institute
Troy, New York

June 1949

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ACKNOWLEDGMENT

The author wishes to thank Professors E. J. Kilcawley, A. A. Burr, and S. Ross for their help and guidance during the experimental work and subsequent evaluation of results.

UNIVERSITY OF MICHIGAN

16-2 Detroit Drive
Rensselaerwyck
Troy, New York
June 2, 1949

Faculty,
Civil Engineering Department,
Rensselaer Polytechnic Institute,
Troy, New York.

Gentlemen:

As partial fulfillment of the requirements for the degree of Master of Civil Engineering, I have the pleasure of submitting this thesis for your evaluation. I hope that the work done in this investigation may in some way be of value to future investigators studying the base exchange properties of clays.

I would like to take this opportunity to express my thanks and appreciation for the knowledge and help that you have given me during my studies at the Institute.

1904 (Gordon) Driver
Hennrichs-Driver
York, New York
June 2, 1904

Family,
1141 (Gordon) Hennrichs,
Hennrichs (Gordon) Hennrichs,
York, New York.

Gordon

As you are interested in the
history of the city of York, I have the pleasure
to send you a copy of the book "The History of York
and its Vicinity" which you will find of great
interest. It is a book of 100 pages and is
bound in leather. It is a very good book and
is a very good book.

I would like to have your opinion as to whether
we should not have a book of the history of York
and its Vicinity. It is a very good book and
is a very good book.

Gordon

PREFACE

Since clay is one of the most bothersome foundation materials, the Soil Mechanics Department of Rensselaer Polytechnic Institute is carrying out a series of inter-related investigations on the properties of clays. This thesis is a small part of that program and was decided upon in an attempt to settle some of the questions raised by Lieutenants W. J. Christensen and William R. Stephenson in their studies of electro-osmosis. The scope is necessarily limited and new limitations were discovered during the investigations. An interesting field of study of base exchange has been opened up, however, and it is felt that some future investigators could learn much by following up this work.

INTRODUCTION

In previous work on electro-osmosis by Lieutenants Christensen and Stephenson, difficulty was encountered in evaluating the results of their experiments because of the many unknowns involved in their clay samples. Knowing nothing of the characteristics of the clay with which they were working, such as the kind of adsorbed ions present, they could not definitely say what changes had taken place.

This uncertainty resolved itself into the problem of obtaining or producing a clay of known characteristics, or better still, a clay with only one kind of adsorbed ion, in large enough quantities to be used in electro-osmosis experiments. Such a clay might be a pure H-clay. With an H-clay, an experimenter might displace the H ions with Mg ions, Al ions, and any other desired ions by using the proper electrode material. The resulting clays could be analyzed for changes in liquid and plastic limits. It might then be determined whether or not stabilization results from osmosis alone or in combination with more strongly adsorbed ions, a theory on which Christensen and Stephenson disagreed.

It was felt that electro-dialysis might be a solution to the practical problem of production. This method would then involve a study of cation exchange on the clay in

question to determine if and when the exchange was complete in order to insure a pure H-clay. With this practical problem in mind, the author undertook this investigation of cation exchange on clays by means of electro-dialysis.

THEORY

The theory of ion exchange on colloids has been known for some time. Helmholtz is generally given credit for determining the mechanism by which such an exchange takes place. The Helmholtz double layer concept in its final form proposes that colloidal particles have adsorbed on their surfaces either a positive or negative layer of ions that results from unsatisfied valences at the broken faces of the particles. This first layer of ions tends to attract by induction a diffuse second layer of oppositely charged particles. This adsorptive force conforms to the equation,

$$F = k \frac{q_1 \times q_2}{d^2} , \quad (\text{figure 1})$$

where q_1 and q_2 are the charges and d is the distance separating them.

If in a stable colloidal solution particles of equal charge and smaller radius than those adsorbed on the colloidal surfaces are introduced, the force of attraction for the new ions will be greater than that for the adsorbed ions and an exchange will take place. This phenomenon is known as "base" or "ion exchange". The rate of this exchange depends in part upon the probability of the new ion getting close enough to the broken surface to effect the change, and thus, upon the concentration of the new ion (figure 2).

As clay is colloidal by definition, it is believed that ion exchange plays an important role where stability is concerned. The stability of a clay is primarily dependent upon its water content. Therefore any method that will control its water content will in some measure control its stability. There are two factors in clay hydration that stand out, its lattice structure and its adsorbed ions.

With respect to lattice structure, there are four main classifications of clays, kaolinite, illite, montmorillonite, and the amphibole-like clays. At the one extreme is kaolinite, whose lattice is divided into two sheets, a tetrahedral silicon sheet and a gibbsite sheet. The contact surfaces of adjacent lattices consist of oxygen ions on the one side and hydroxal ions on the other. The strong attraction of these ions makes this lattice a tightly knit unit very difficult to break down or to penetrate with water. At the other extreme is montmorillonite, whose lattice is composed of a tetrahedral silicon sheet, a gibbsite sheet, and another tetrahedral silicon sheet. The contact surfaces of adjacent lattices in this clay consist of oxygen ions alone, giving a much weaker bond that allows water to penetrate between lattices and decomposition to take place. The other classes of clays fall somewhere in between these two.

Since ion exchange is a surface phenomenon, one can see that the exchange capacity of montmorillonite is much greater than that of kaolinite, due to the greater surface area available. The loose structure of montmorillonite even allows ions to penetrate within the voids of the lattices. It is this exchange capacity that we are interested in as a means of stabilization.

In order to understand how this exchange and stabilization would take place, we should understand ionic hydration. A cation, for instance, becomes hydrated when in solution due to its attractive force for dipolar water molecules (figure 3). Hans Jenny is given credit for the discovery that smaller ions hydrate to the greatest extent and, as a result, end up with the largest hydrated radius is the determining factor when force of adsorption is considered, the ionic charge being the same. Jenny formulated the lyotropic series which gives the order of adsorption and release of ions as follows:

Adsorption: $\text{Li} < \text{Na} < \text{K} < \text{Rb} < \text{Cs} < \text{H}$

Release: $\text{Li} > \text{Na} > \text{K} > \text{Rb} > \text{Cs} > \text{H}$.

A similar series exists for bivalent and trivalent ions. It may be seen that attraction is in reverse order of dehydrated ionic radius except for hydrogen. This series is disturbed in some clay systems by the tendency of certain ions to dehydrate. The hydrogen ion is a special

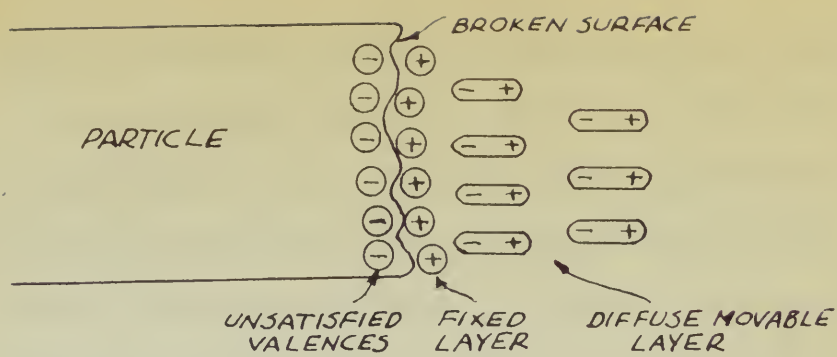


FIGURE 1

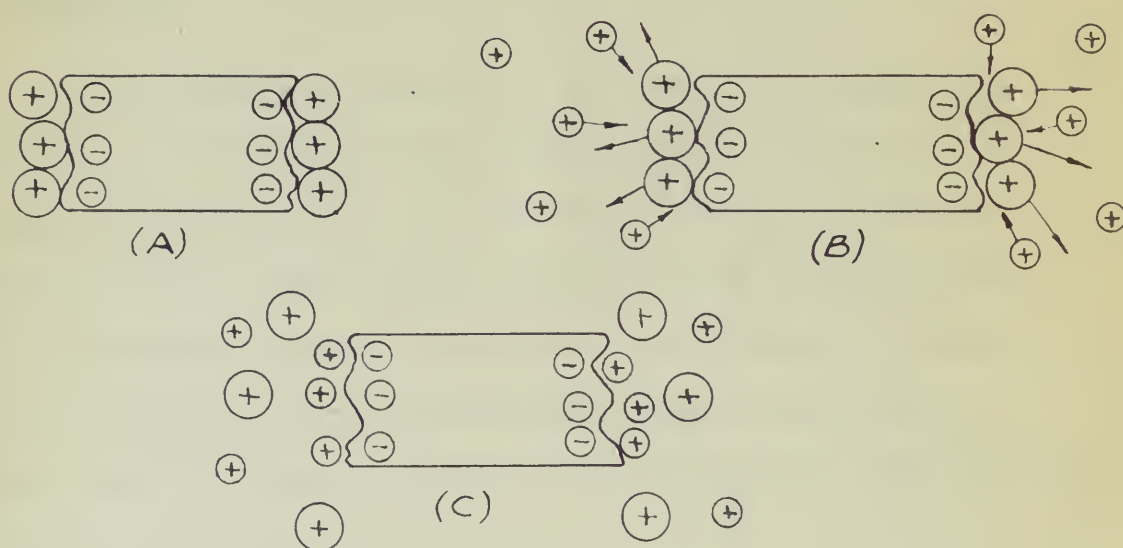


FIGURE 2

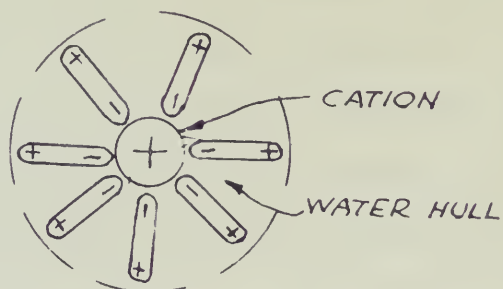


FIGURE 3

case in that it behaves contrary to the hydrated radius theory. It is generally accepted that the adsorption of the hydrogen ion is more chemical than electrostatic. Instead of being separated from an adsorbing hydroxal ion by a water film as a calcium ion would be, the hydrogen ion actually meshes into the electron orbit of the hydroxal ion (figure 4). In the process of exchange then, and in exchange alone, the hydrogen ion acts as though dehydrated.

The problem of producing an H-clay is intimately tied up with the stability and exchange capacity of clays. Since the hydrogen ion is the most strongly attracted ion of all, we need only to supply these hydrogen ions in sufficient quantity to permit the exchange to take place, and to remove the replaced ions to facilitate more rapid exchange. This is the function of the dialysis cell.

The theory of such a cell is simple. The cell consists of three compartments separated by membranes porous only to ions. Electrodes are inserted in the two end compartments (figure 7). The clay solution is placed in the center compartment and water for receiving the displaced ions is placed in the end compartments. When a voltage is applied, the following action takes place. Dissociated hydrogen ions in the center compartment replace the adsorbed cations (figure 5A). These cations are at-

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tracted to the cathode and will pass through the membranes, though the clay solution will not. The leftover hydroxal ions are attracted to the anode (figure 5B). If distilled water is used in all compartments and there is no electrolysis, this action continues until the cation exchange is complete. If electrolysis takes place, the water that breaks down has the following effects. The hydroxal ions at the anode give up electrons and oxygen is released. The hydrogen ions at the cathode receive electrons from the cathode and go off as hydrogen gas (figure 6). This results in an acid anolyte and an alkaline catholyte. The process is somewhat more complicated when distilled water is not used.

There are some theories that say the lattice structure of a clay is broken down or possibly changed by electrodialysis. Any such change may be detected by a study of diffraction patterns. Any particular lattice structure has the property of diffracting X-rays in a pattern peculiar to that one structure. If any change takes place in the lattice structure, this change may be detected by a change in the diameters of diffraction rings recorded on X-ray film.

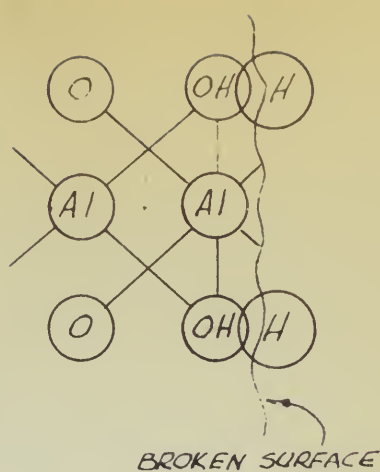
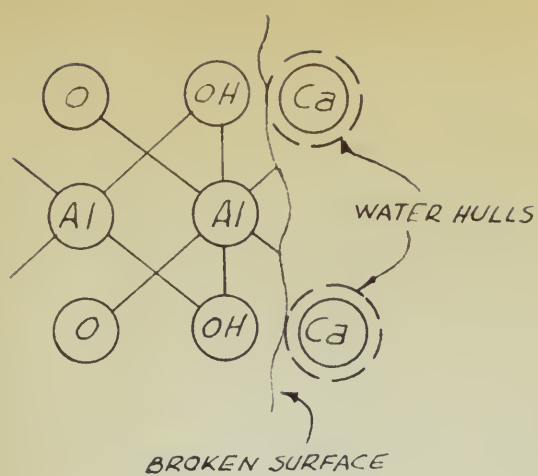


FIGURE 4

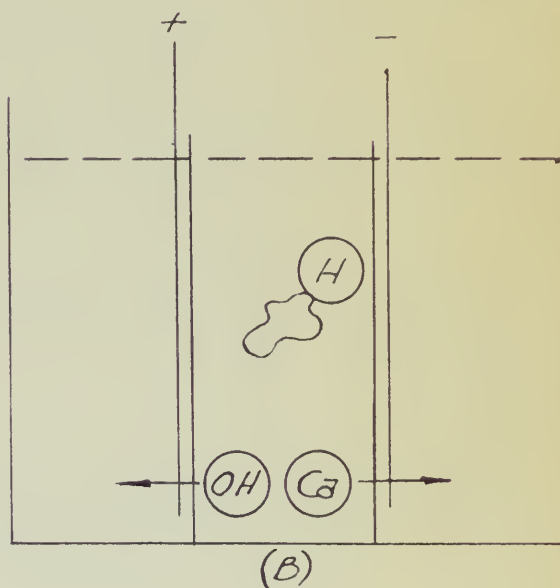
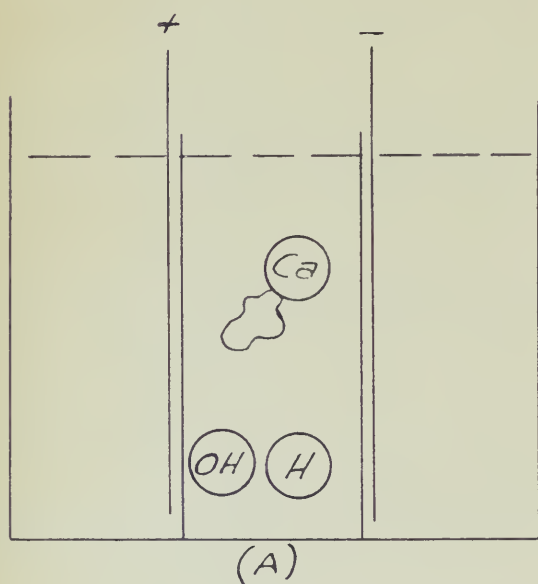


FIGURE 5

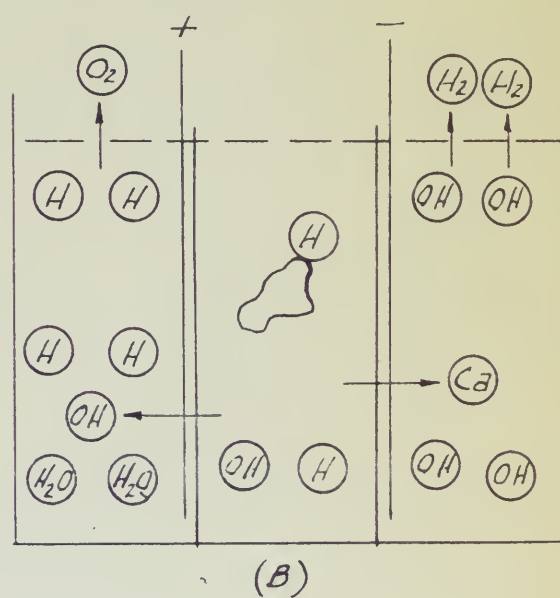
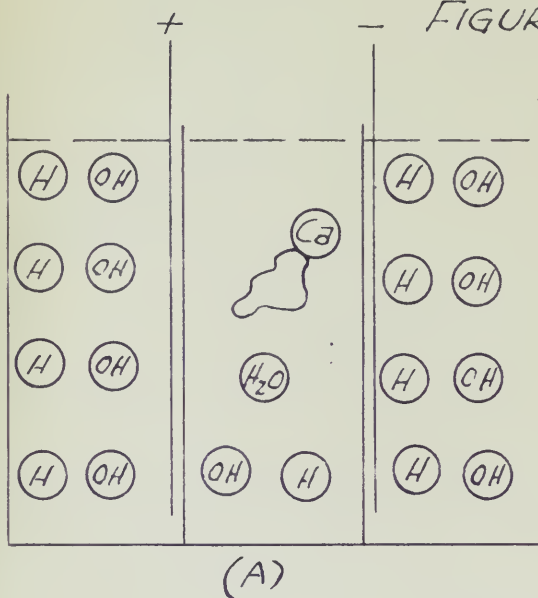


FIGURE 6

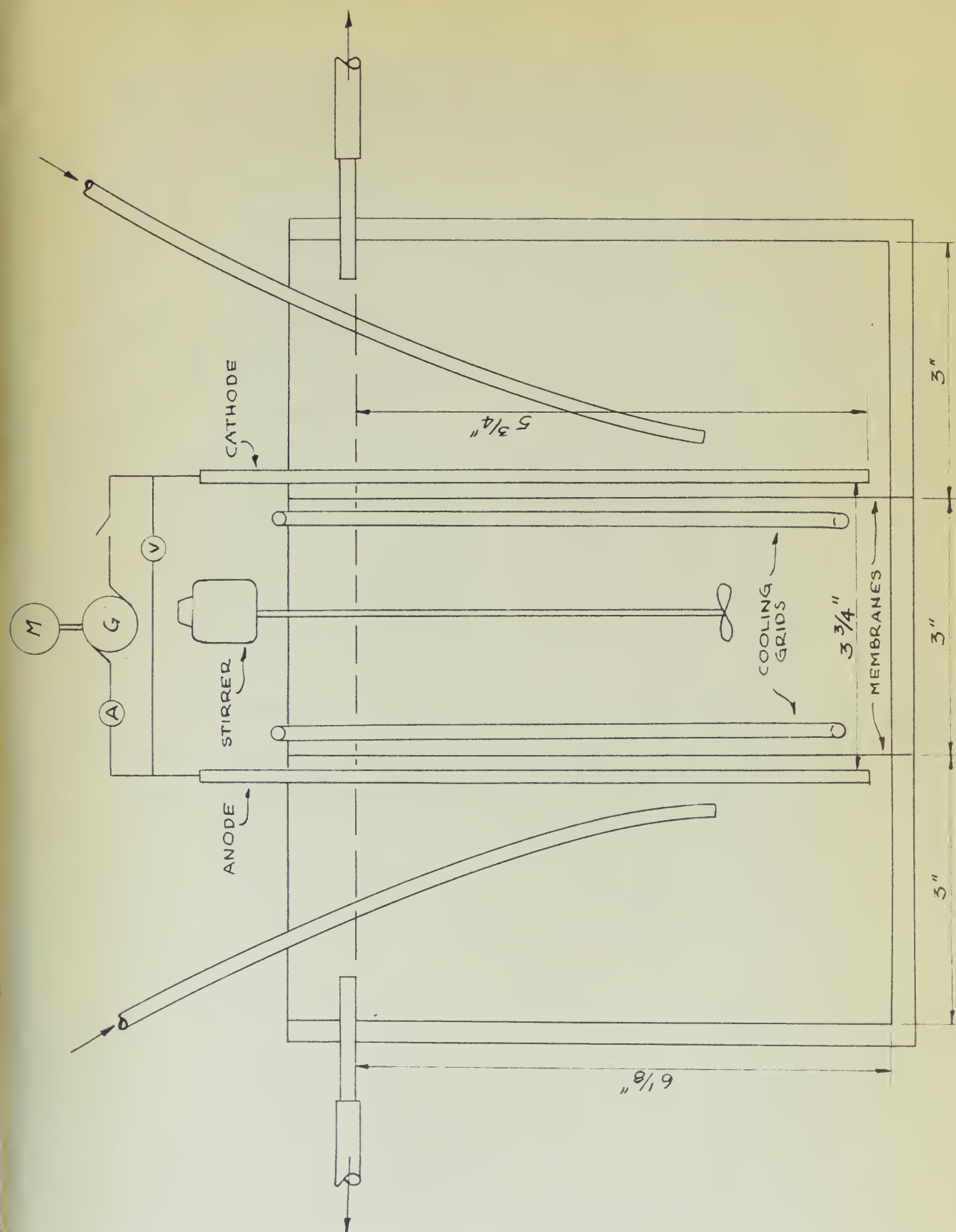


FIGURE 7

APPARATUS

The following equipment was used:

- a. 1000 watt, 120 volt gasoline driven d.c. generator (figure 8).
- b. Dialysis cell made from a storage battery case (figure 9).
- c. Carbon electrodes, submerged area = 31.6 inches².
- d. Monel metal electrode, submerged area = 23.6 inches².
- e. Variable speed stirrer.
- f. D.C. voltmeter, 0-150 volts.
- g. D.D. ammeter, 0-30 amperes.
- h. Beckman pH meter.
- i. Standard X-ray diffraction equipment.

All experimentation was done in the soil mechanics laboratory on Avenue B and the Ricketts building at Rensselaer Polytechnic Institute, Troy, New York.

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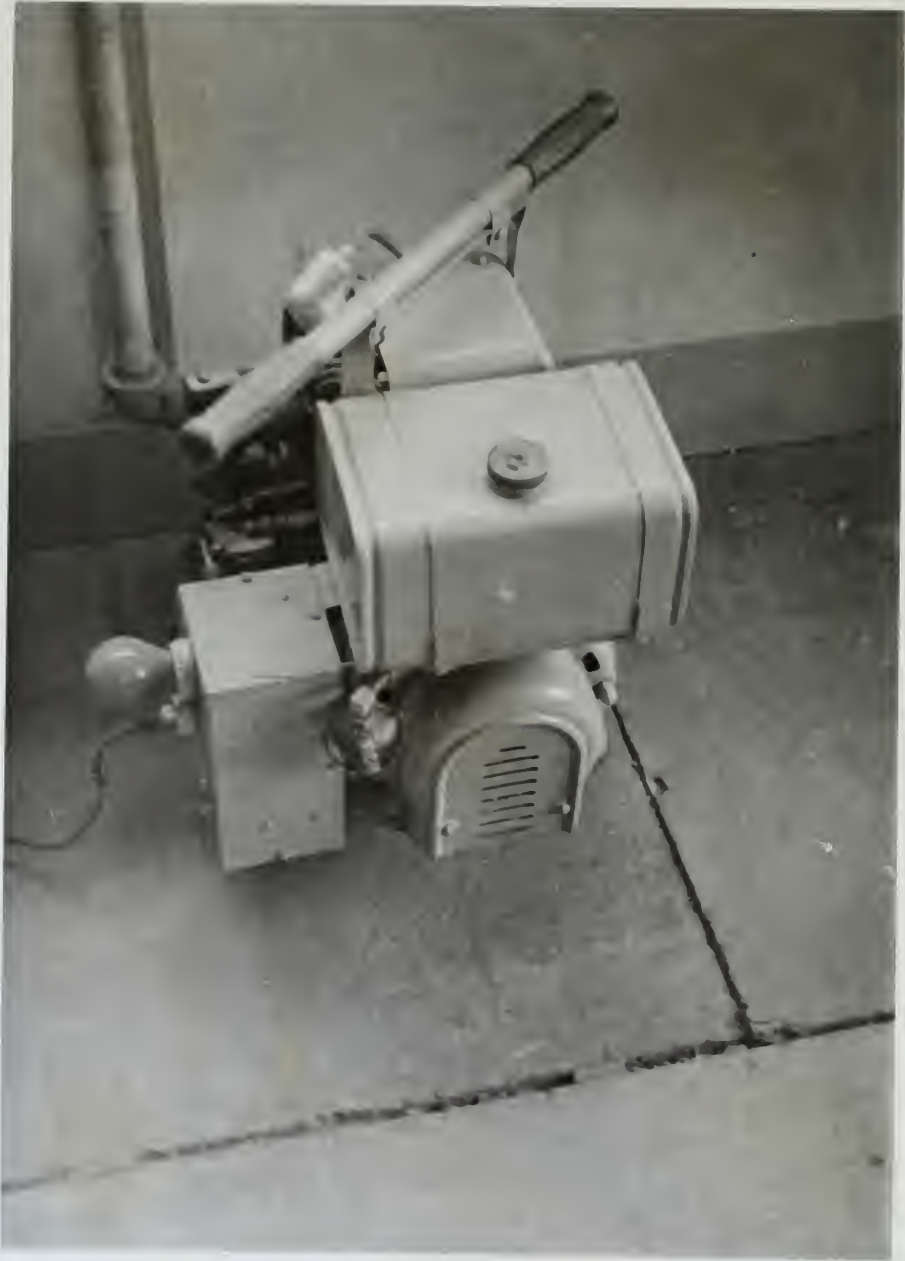


FIGURE 8

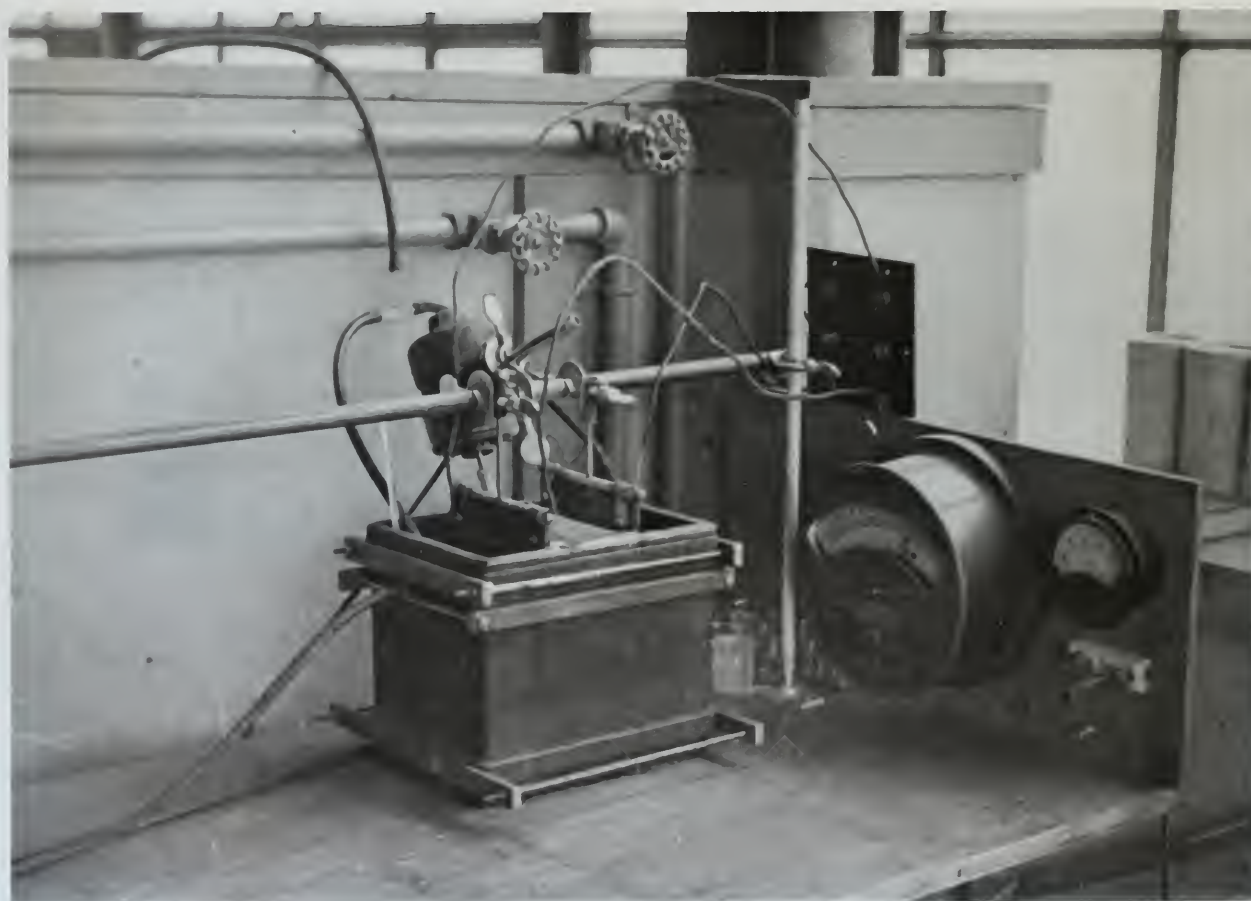
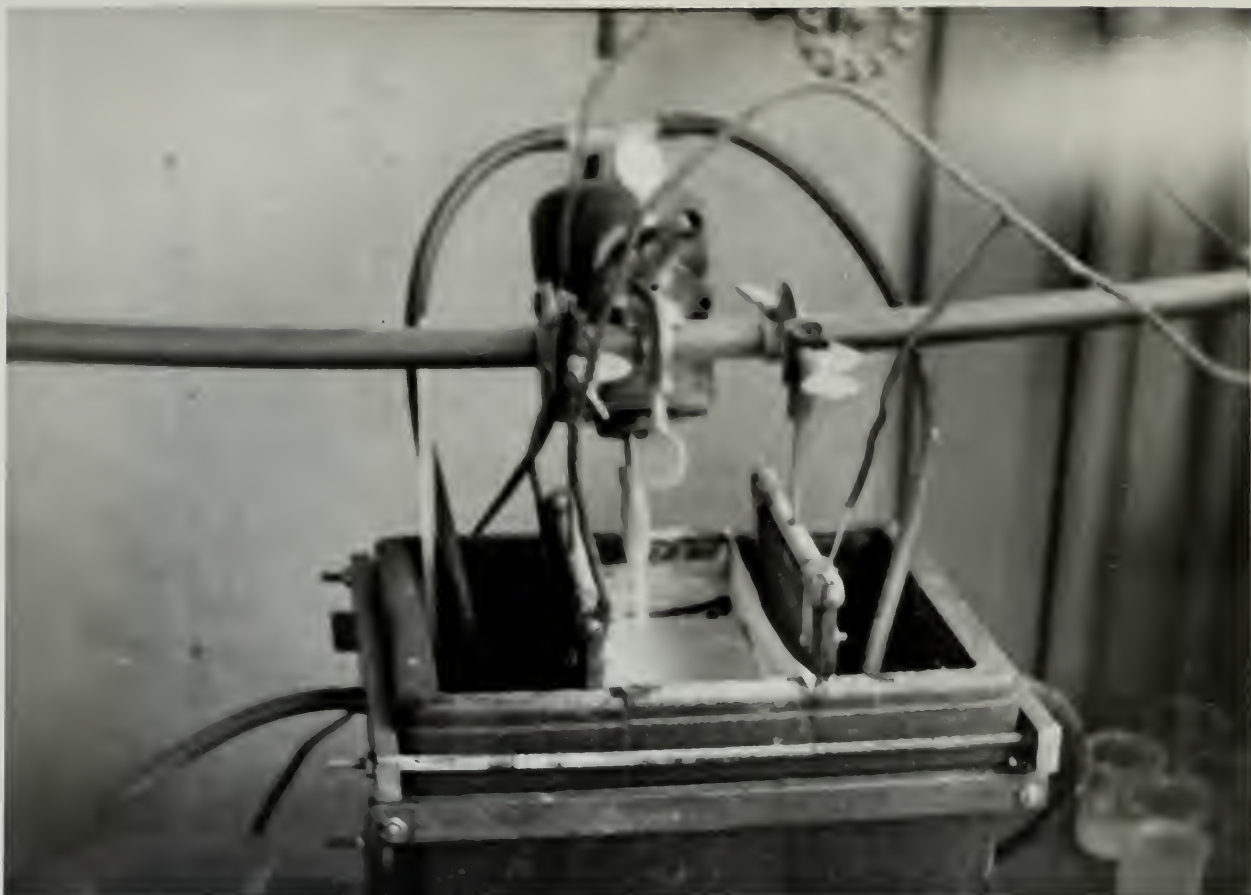


FIGURE 9

PROCEDURE

The procedure for the several runs varied somewhat due to discoveries made in previous runs. In all cases the clay was prepared by grinding it very fine and then making a sieve analysis. In most cases 200 grams of clay was used.

The cell used in each case was one of two made by the author (figure 9). They were made from a standard automobile storage battery sawed into three equal segments along the longest dimension. Cell #1 had four holes drilled longitudinally in the walls to receive the bolts used to clamp the segments together. Rubber gaskets were used between the segments to make the cell watertight. Cellophane membranes were used in every case and were placed so as to separate the segments. When assembled the cell consisted of three distinct watertight compartments adjacent to the membranes, spaced about 3 3/4 inches apart. The power source of 120 volts was connected across these electrodes. This cell was used in Run #1 only.

Due to difficulties encountered in Run #1, this first cell was modified by adding influent and overflow pipes to each of the two end compartments to provide for constant circulation of the dialytes. This cell was used until a leak developed when Run #3 was begun. As

the leak could not be repaired, a new cell was made which differed but slightly from cell #1. Instead of using gaskets, and running tie bolts through the cell walls, the segment faces were sanded to watertight smoothness and external clamps were used. This cell was used for the remaining runs.

In each run a predetermined amount of water was added to each compartment so that all water levels would be the same. The clay was added to the center compartment where it was constantly agitated by a stirrer.

During the runs a record was kept of the temperature of the dialytes, the current, the voltage, and the pH values of the dialytes. In each case the run was continued until the pH indicated that ion exchange was complete or until adverse conditions made it necessary to stop.

In every case except Run #4 an X-ray diffraction pattern of the clays was obtained before and after treatment to determine whether or not the lattice structure had been changed.

RUN #1

Cell #1 was used for this run with no circulation of the dialytes. In each of the two end compartments 1000 cc of distilled water was placed. In the center compartment 925 cc of distilled water and 200 grams of an unknown clay were placed so as to give 1000 cc of solution. Subsequent analysis by X-ray diffraction proved this clay to be primarily attapulgite. This was more or less borne out by a differential thermal analysis that indicated the clay was different from kaolinite, illite, and montmorillonite. A monel metal anode and a carbon cathode spaced 3 3/4 inches apart were used in this run.

This first run was to determine what ions would be removed in order to see what chemical tests would be necessary in determining the order of base exchange. This would eliminate making tests at the various time intervals for ions that were not present.

The 120 volt source was applied across the electrodes and immediately there was evidence of a gas being liberated at the cathode. The pH began to climb quite rapidly at the cathode and to decrease at the anode. This change continued for the first 4 hours, after which the pH levelled off, a phenomenon that was to have been used as an indication of completion of base exchange. Treatment was continued, however, for another 20 hours and the pH remained substantially the same at both electrodes.

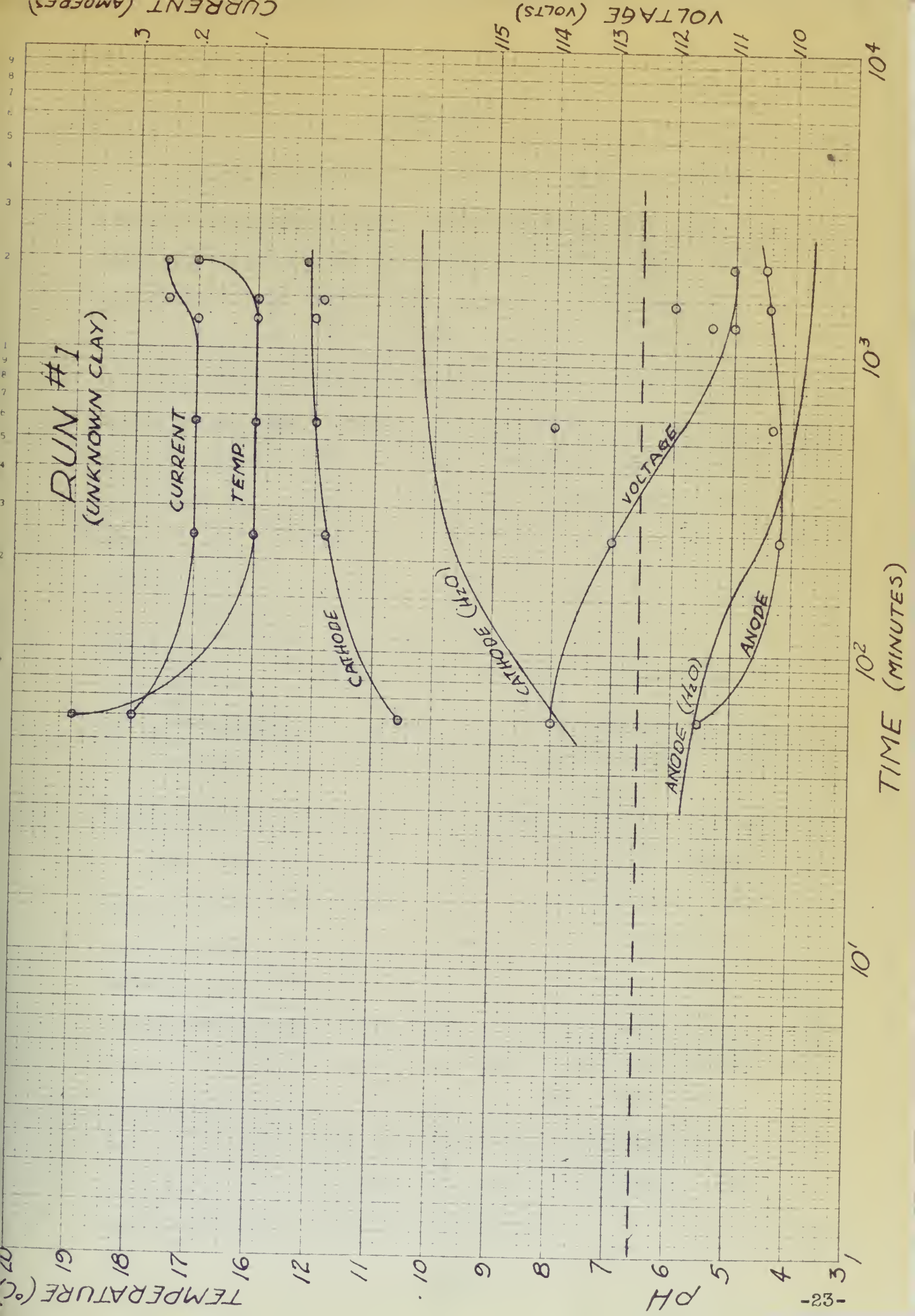
After some 8 hours of treatment, the clay began to pack up on the anode membrane and a green precipitate began to form at the anode. The clay was periodically scraped from the membrane to keep it from clogging and to provide better dispersion. The effect of the clogged membrane would have been an increase in the resistance to ion flow. The green precipitate was identified as the hydroxides of copper and nickel, indicating that the anode was being attacked by an electrolysis process.

The excessive alkalinity and acidity in the end compartments and the constant pH values after only 4 to 8 hours of treatment indicated that the procedure was not correct. Allowing the exchanged ions to accumulate in the end compartments caused an osmotic pressure to build up sufficiently to stop the base exchange. A condition of equilibrium had been established but the base exchange was not complete.

The dialytes were given a qualitative chemical analysis to determine what ions were removed. It was found that calcium was definitely present in the anolyte, a surprising development. There were traces of aluminum, magnesium, and calcium in the catholyte. This meant that the clay was predominant in calcium as nearly as could be determined from this run. It can reasonably be assured that the presence of calcium at the anode indicated a slight breakdown of at least some of the clay. This is the only way in which calcium could appear at the anode, as a complex ion, instead of at the

cathode as a simple calcium ion. It is felt by many that this does occur with the montmorillonites and amphiboles.

A sample of the clay was taken for diffraction pattern purposes. These patterns were subsequently lost, as were those for Run #2, by a photograph processing laboratory before measurements could be made to determine whether or not there was any difference between the patterns before and after treatment.



RUN #2

In order to alleviate the osmotic pressure resulting from too high an ionic concentration at the electrodes, it was decided to circulate the dialytes. Distilled water would have been the most satisfactory medium in order that the dialytes could be analyzed qualitatively, but a three-day supply was not available. Tap water was therefore used. An overflow pipe and an influent tube were added to each compartment and water was circulated at as fast a rate as the overflow pipes would take.

It was thought that a greater dilution of the clay would do something to prevent the electrophoresis of the particles by allowing more to remain in solution. For this reason the amount of liquid was doubled in all compartments. All other conditions were the same as in Run #1 and the same procedure was followed.

Much the same thing happened as in Run #1 except for two noticeable things. The current rose to a much higher value, but this was attributed to the changed set-up. The green precipitate began to form in large quantities and at a rapid rate. This was surprising, since it was felt that the circulation of the dialytes would prevent this. At the end of eight hours, however, the anode overflow pipe clogged and caused the anolyte to flow over into the center compartment. The run was stopped and the anode removed.

At least half of the anode had been corroded completely away by the electrolytic action. Inspection of the metal stirrer revealed that it was badly pitted and the propeller had corroded away. Because of these actions, some more changes were made for this run.

To prevent further corrosion it was decided that carbon would be used for the anode as well as the cathode. A glass stir rod was substituted for the metal one. With these changes made, Run #2 was continued as before.

A change in technique had also to be made. With circulating water in the end compartments it was thought that a return to normal of the pH values of the dialytes, that is their value before treatment was begun, would indicate no further removal of adsorbed ions. It was apparent, however, that electrolysis was playing a very important part in the process. For this reason a new indication of completion of ion exchange had to be found. It was felt then that for a constant voltage there would be a constant rate of electrolysis, assuming no change in the make-up of the circulating water or its rate of circulation. The pH readings should return, therefore, to a value that was dependent upon electrolysis alone. When the pH readings levelled off, ion exchange should be complete.

Since tap water was being circulated, however, no chemical analysis could be made to see what ions

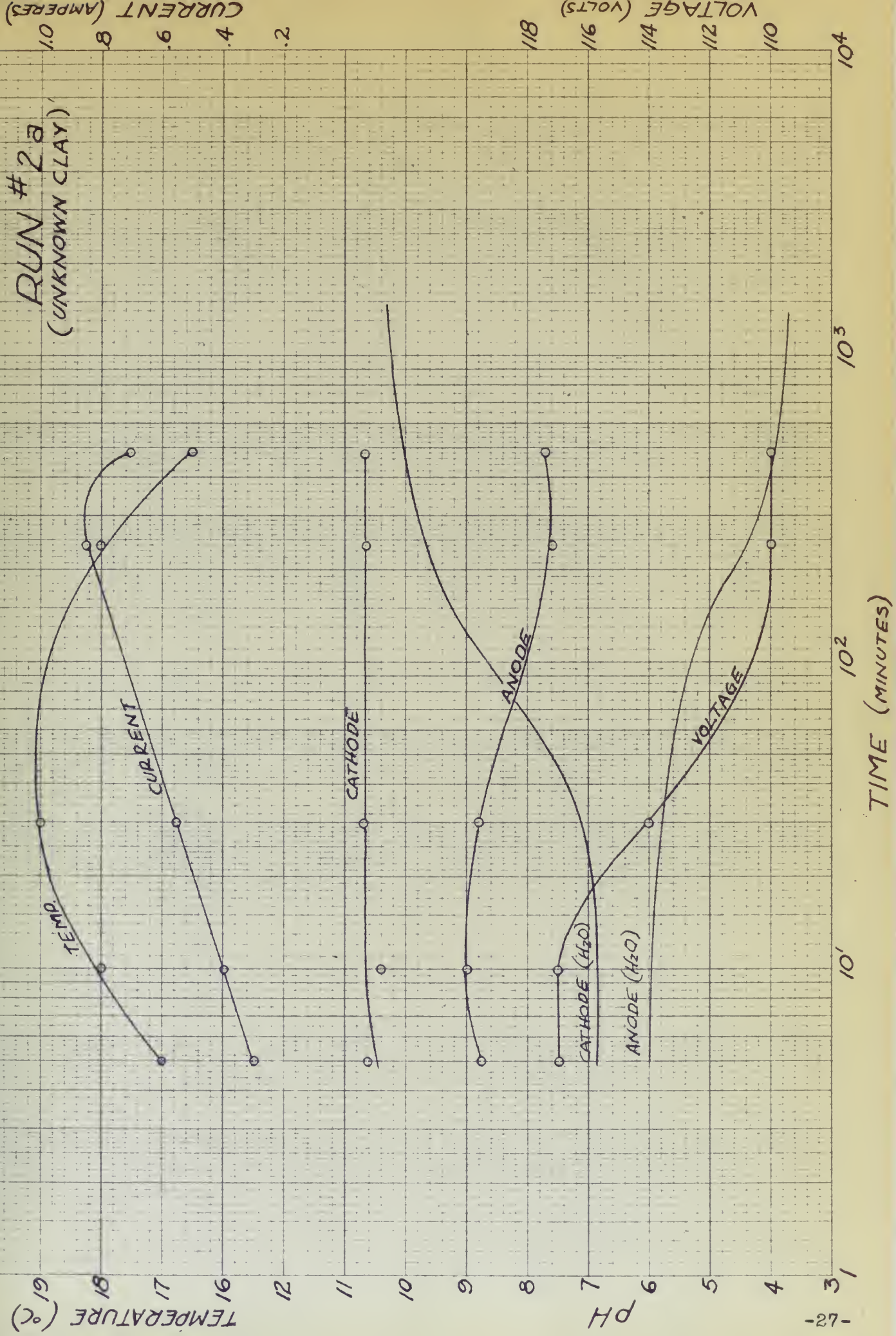
were being removed and in what order. For this reason samples of the clay were removed at various times to be analyzed, if possible, by spectrographic means.

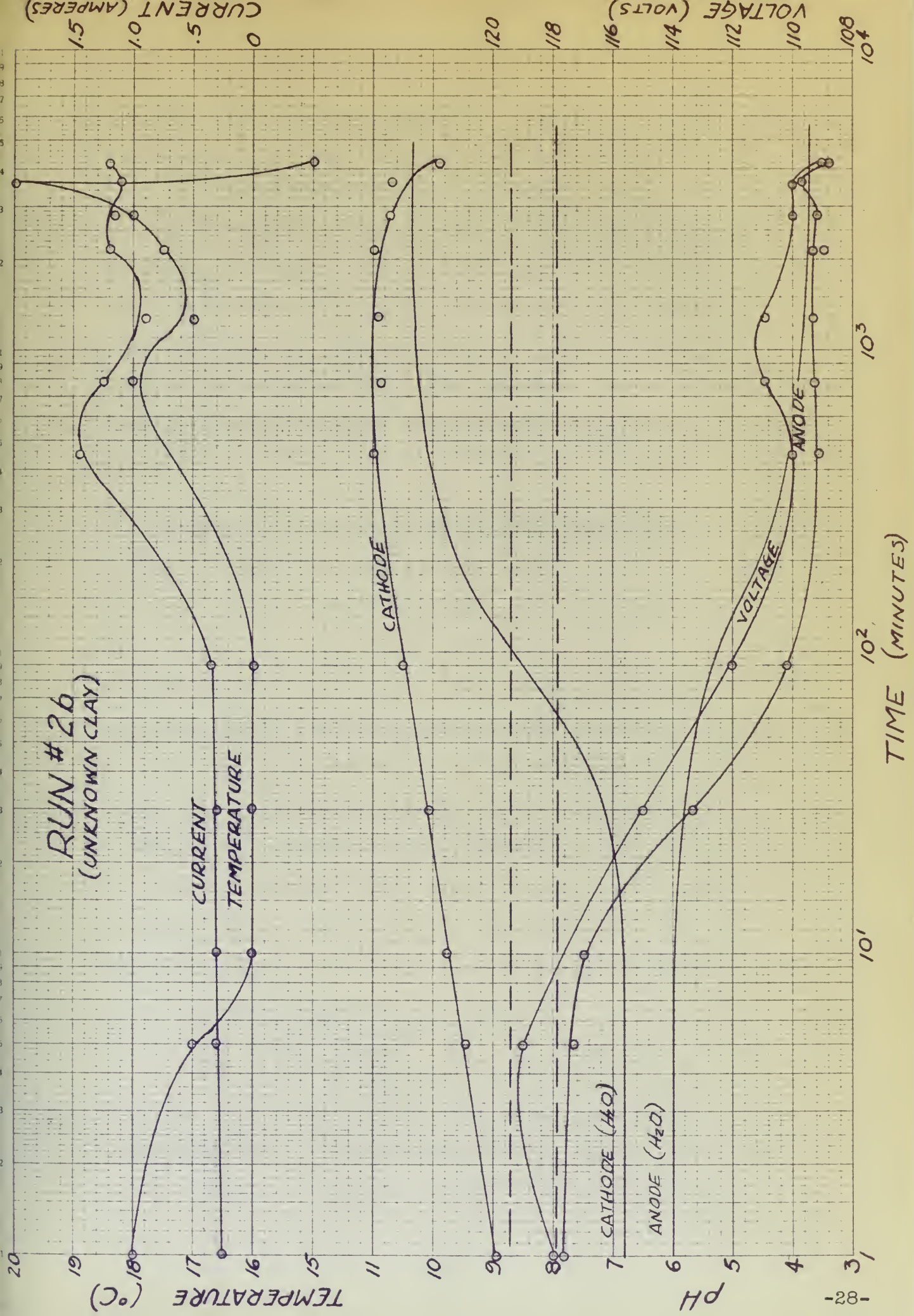
The run was continued for approximately 70 hours when the pH readings indicated that exchange was complete. This would have to be substantiated, time permitting, by further analysis. Nothing new developed in this run. Gas was continuously emitted at both electrodes. Clay did pack up on the anode membrane again due to electrophoresis. Some other method would have to be devised to prevent this.

After this run it was noted that the anode began to take on a spongy appearance. The surface was somewhat soft and powdered carbon could be rubbed off. Apparently the anode was being oxidized, as some forms of carbon are known to oxidize under similar conditions.

X-ray diffraction samples were again obtained after treatment but their patterns were lost as previously mentioned.

TEMPERATURE (°C)





RUN #3

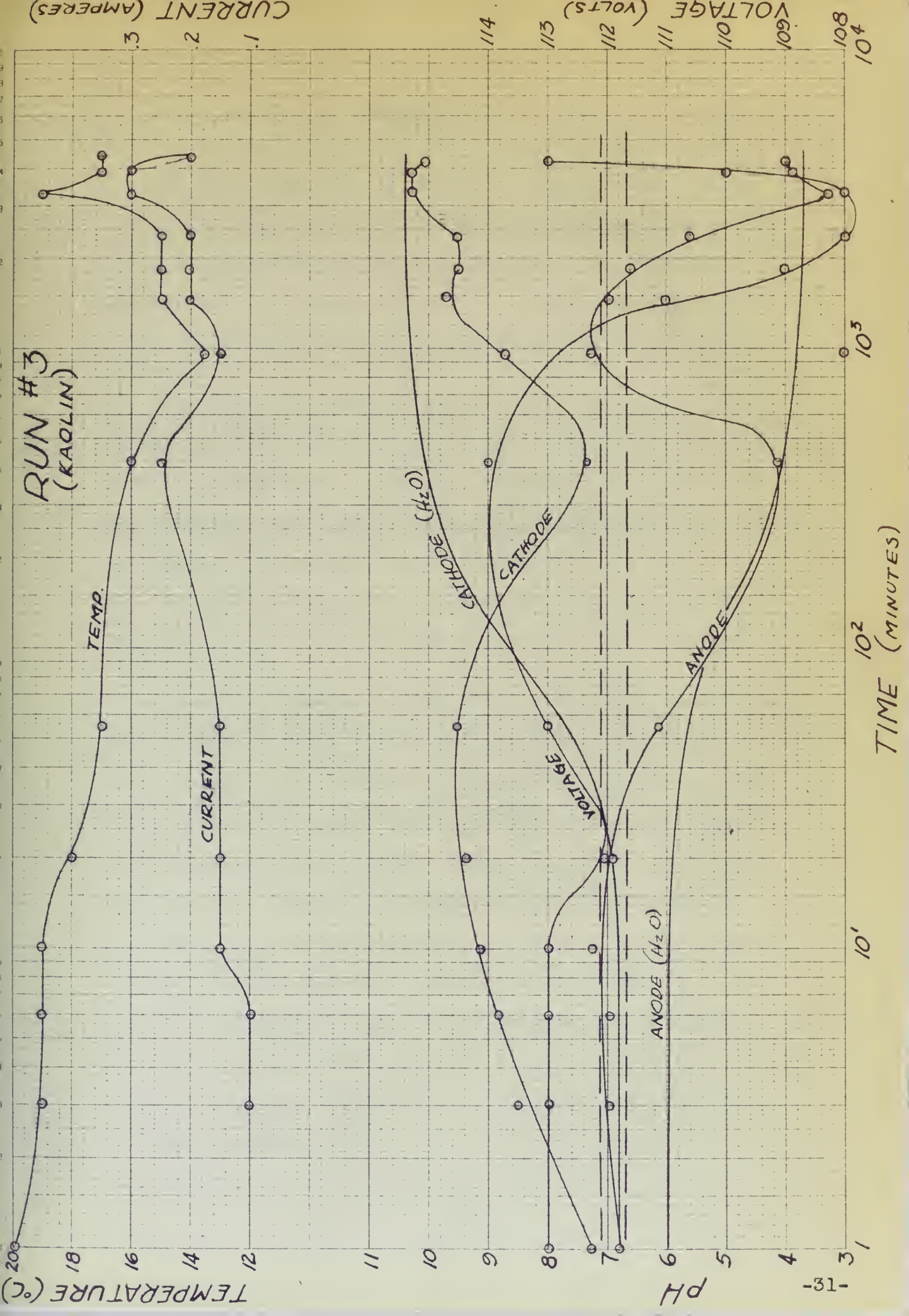
In order to compare the different classes of clays in regard to ease of conversion to an H-clay and ionic exchange in general, this run was made using kaolin. It was at this time that a leak developed in the cell and a new one was made after several attempts to stop the leak in the original one. The procedure did not vary from that used in Run #2 and the same electrodes were used.

As usual gas was evolved at both electrodes and clay had to be removed from the anode membrane at regular intervals. At one time during the run the circulating water supply was cut off by accident so that a condition similar to Run #1 existed. This was apparent in the pH readings. This condition may have been responsible in part for the large deviation in the pH trend that followed. It is believed, however, that variations in the output of the generator were largely responsible for the oscillating pH curves that were obtained. This can be assumed, since electrolysis is proportional to the applied voltage and the major part of the pH value is thought to be a direct result of electrolysis.

A plot of the pH readings was no surprise, as it revealed the kaolin to have less exchange capacity than the attapulgite. This was evidenced by the lesser slopes of the curves, indicating a lesser tendency to give up

adsorbed ions and/or fewer adsorbed ions. The reason for the "and/or" is that complete removal, as indicated by the downward pH trend at the cathode, took the same length of time as for attapulgite. If there were only fewer adsorbed ions, they should have been removed sooner, unless rate of exchange is just as dependent upon the concentration of the adsorbed ions as it is upon the concentration of the substituted ions.

After approximately 70 hours, when the pH trend indicated complete exchange, the run was stopped. A sample was again taken for X-ray diffraction purposes. A comparison of the diffraction patterns before and after treatment showed a definite increase in the diameters of the diffraction rings. The most probable explanation of this would be that the lattice structure, though not changed, was strained as a result of some part of the exchange process.



RUN #4

To follow the same line of reasoning that was used in Run #3, an Osage, Wyoming, bentonite was used for this run. As previously mentioned, bentonite (a montmorillonite) is the extreme case of a flexible lattice structure. This was evidenced by what followed in this run.

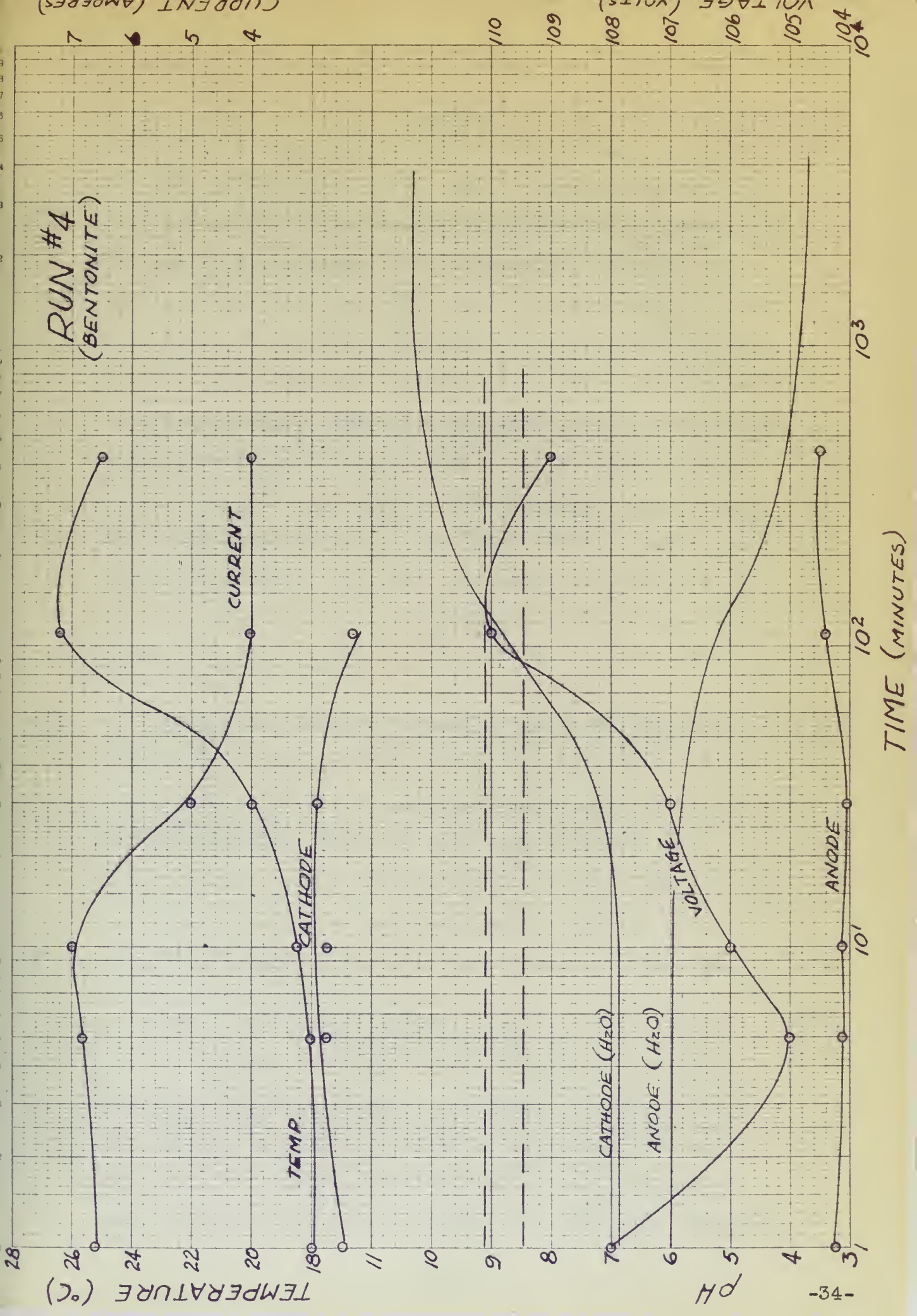
The clay was added to the water in the center compartment and it became apparent that some difficulty would be encountered. The clay proved to be thixotropic and except for the area directly adjacent to the stirrer, the center compartment was full of a gel. As bentonite is very expansible it was evident that too much had been used, but it was decided to see what would happen.

When the voltage was applied, the current immediately rose to 6.7 amperes, almost thirty times the average initial value. At the end of one minute this value had risen to 8 amperes, and the pH at the anode and cathode was off the Beckman pH meter scale, 0 in the first case and 13 in the latter. The current rose to 10 amperes and the clay gel began to bubble. There was much gaseous activity at the anode and cathode. Steam began to rise from the gel which had reached a temperature of 75 °C. The clay was now liquid adjacent to the stirrer and the cathode membrane. Because of the excessive heating the run was stopped.

Part of the gel, equivalent to 28 grams of clay, was removed and an equal amount of distilled water was added. Two cooling grids were placed in the center compartment through which tap water was circulated in an effort to keep the temperature down.

With these changes made, the run was started again. Much the same thing happened as before, except that the pH, temperature, and current were enough lower than before, it was thought, to continue the run. The same gas evolution and bubbling of the gel took place with no new developments. At the end of 7 hours, however, it was apparent that there was a leak in the cathode membrane. This was due, no doubt, to embrittlement of the membrane caused by the excessive heat. The cathode membrane in all runs ended up brittle to a certain extent, while the anode membrane remained pliable. This would indicate that some other membrane material might be used to advantage on the cathode side.

In order to cut down on the ion exchange and , consequently, the current and temperature the run was begun again using a new sample of 100 grams of bentonite. The same high values were recorded for the first 30 minutes, after which time the membrane ruptured again. In all three trials clay packed up on the anode membrane to a much greater degree than in any of the previous runs. The attempt to make an H-bentonite was abandoned in order that more work could be done on the attapulgite.



The purpose of this run was to see exactly what effect the particle size had on the rate of ionic exchange. Not all of the effects of an ultrasonic vibrator are known, especially on clays. The vibrations seem to have the effect of dispersion and, at the same time, of flocculation. In this run a 2000 cc solution with 200 grams of attapulgite was subjected to vibrations at 1000 kilocycles per second for one minute. After the vibration some of the clay had settled very compactly in the bottom of the container while the rest formed a very homogeneous solution. The same electrodes were used as in previous runs and the same procedure as for other attapulgite runs was used.

One of the effects of the vibration was apparent immediately as the pH value at the cathode indicated a faster rate of exchange than had previously been noted with attapulgite. What is more, the pH climbed to a higher value and the current was generally higher. This was to be expected, since the rate of exchange is a function of surface area and the vibrations gave the necessary increased dispersion.

The second, and most important and interesting, phenomenon resulting from the vibration was that at no time did the clay have a tendency to pack up on the membranes. A possible explanation is here submitted. When electrophoresis takes place, the negatively charged

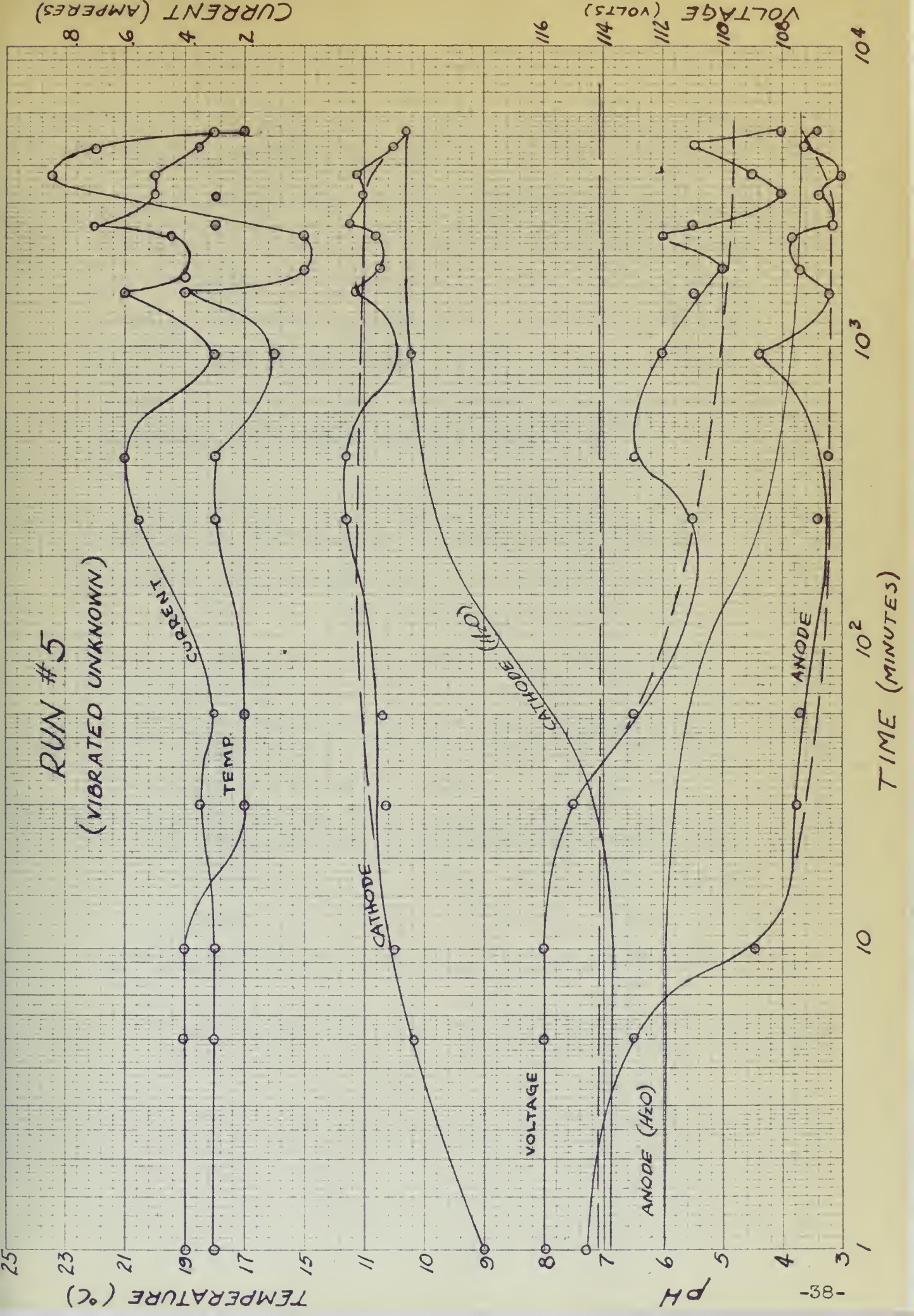
clay particles are drawn to the anode. The particles are large enough (force of repulsion of two particles is inversely proportional to the distance between them squared) so that the natural cohesive forces overcome the repulsive forces and the clay packs up on the anode membrane. But when the clay is vibrated, it is broken down into particles small enough so that the repulsive forces overcome the cohesive forces and the particles remain in solution instead of packing up on the membranes. There was a tendency for more clay to settle to the bottom of the cell also, a result perhaps of the flocculation effect of vibration.

At two points in the run the motor-generator set cut out and was off about an hour. This had the effect of starting the pH values at the electrodes back to normal. Other than these two events, the run proceeded normally until the fourth day.

When the 4570 minute reading was taken it was noticed that the level of the liquid in the center compartment was much lower than that in the end compartments and clay was appearing at the cathode. This was very puzzling since a leak in the cathode membrane should have resulted in the same liquid level in the two connected compartments. There was also an extreme difference in the two pH values. It was felt that the clay may have been decomposed finely enough to penetrate the pores of the membrane.

In order to determine the cause of this phenomenon the run was continued to see what would happen. The next morning, however, the three liquid levels were the same so the run was stopped. An inspection of the membrane revealed a leak of the usual type, due to embrittlement. The difference in liquid levels could be explained only by the presence of some force moving the solution from the center compartment against the hydrostatic head until the hole became too large.

Samples of the clay were taken for X-ray diffraction analysis after vibration and before treatment, when the leak first developed, and when the run was completed. A comparison of these diffraction patterns again indicated a trend of increasing diameters after treatment, though not so pronounced as for kaolin. This is perhaps explainable. A diffraction pattern is the average pattern for all of the lattice structures present in the sample. Kaolin has a definite structure with a stable fixed thickness. A straining of this lattice would, therefore, be rather noticeable. On the other hand, attapulgite has a looser lattice structure with a thickness that varies from one to two times its smallest value. The clay particles in a diffraction sample might have lattice thicknesses throughout this whole range, and the diffraction pattern is an average for all of these lattices. Any straining of the lattices in this case would not show up to such a large degree. It is felt that some straining of the lattice structure did take place.



RUN #6

Before it could be determined exactly how much of the pH change was due to ionic exchange, the effects of electrolysis of the water had to be determined. It was decided, therefore, to make a run using distilled water alone in the center compartment. Keeping in mind that the water undoubtedly would not react the same without clay as it did with clay, the author felt that a fair comparison could nevertheless be made.

The same set of electrodes was used and the same procedure was followed throughout the run. As had been expected, the pH values were slow to change and to reach the maximums. The unexpected upward trend at the anode could not readily be explained. It was also noticed that the anolyte was slightly turbid, though the catholyte was not, and remained so for almost 2 hours, at which time the pH began to fall.

The motor-generator set cut out again and was off approximately an hour and a half. Other than this, the run proceeded very normally. After some 48 hours the pH values levelled off and remained that way for the next 24 hours, at which time the run was stopped.

TEMPERATURE (°C)

RUN #6
(DISTILLED WATER)

TEMP

CURRENT

CATHODE

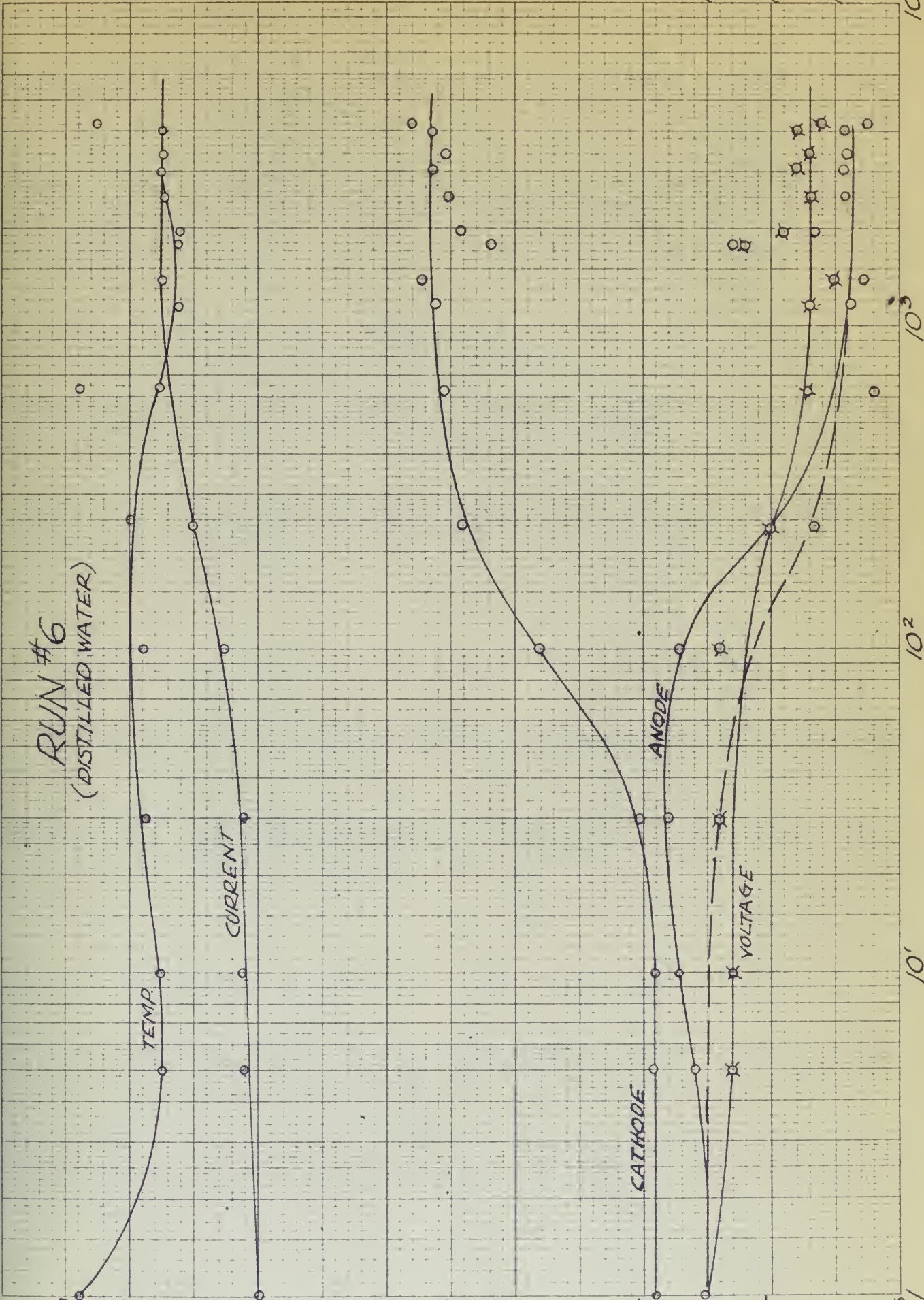
ANODE

VOLTAGE

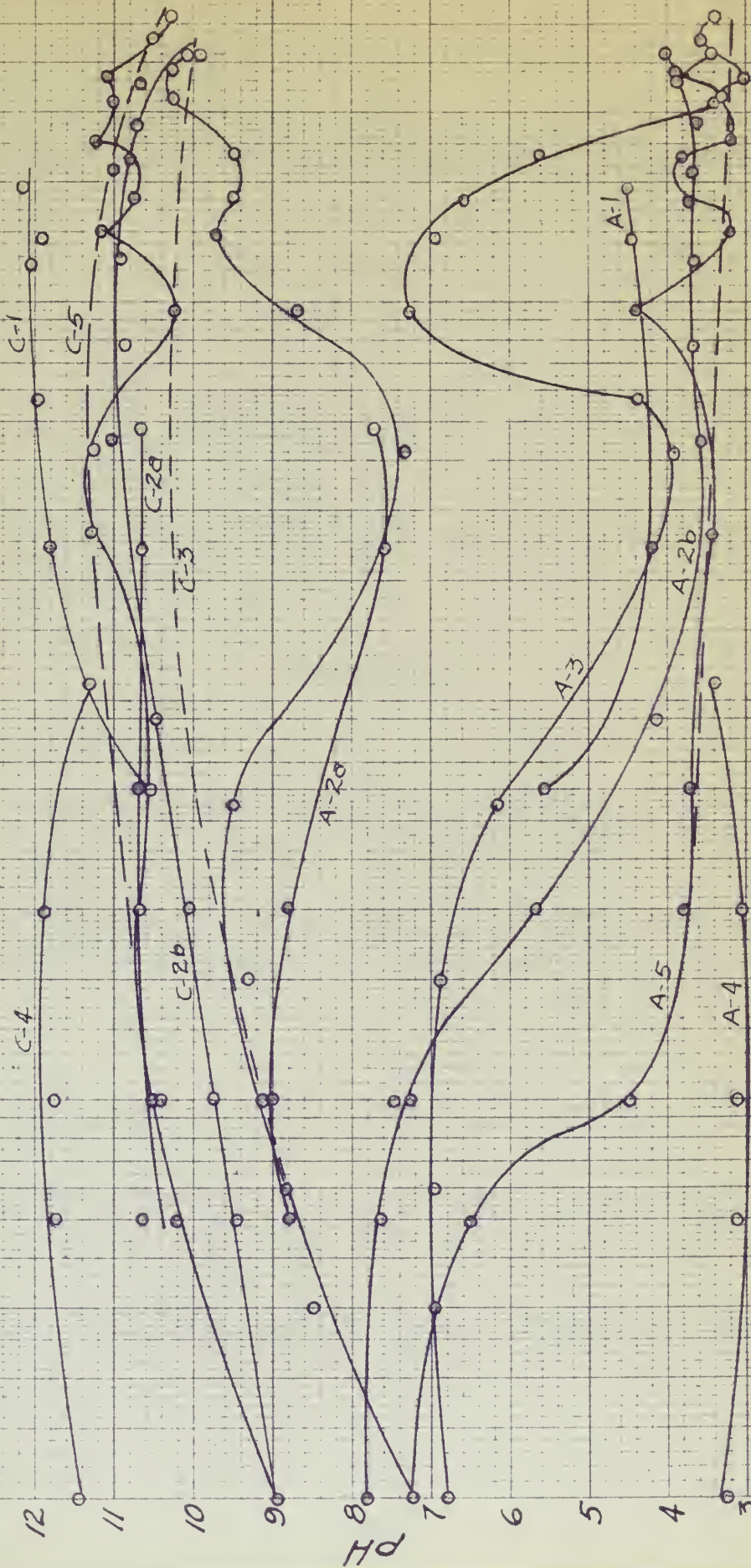
pH

VOLTAGE (VOLTS)

TIME (MINUTES)



COMPOSITE OF pH PLOTS



CONCLUSIONS

CONCLUSIONS

CONCLUSIONS

Despite the many variables and limitations in an investigation of this sort, quite a few rather definite conclusions may be drawn from it. As an investigation of the practical problem of producing a pure H-clay for further use, it has been shown that such a conversion from an unknown clay to an H-clay can be effected. The definite return of the pH at the cathode to a known value due to electrolysis alone indicates rather decidedly that ionic exchange has been carried to completion. Definite proof of this theory was obtained by the spectrographic analysis made by R. F. Reuss in his work on spectrographic analysis methods of identifying clays on the sample used in Run #2b. The disappearance of the calcium line indicated that calcium had been removed from the clay. Its appearance again after treatment was probably due to a mistake in procedure. The final sample of clay was taken from the cell the day after the run was completed. This allowed the calcium and magnesium in the tap water to pass through the membranes by dialysis and into the clay mix. By this process the percentage of calcium and magnesium increased. More accurate results could have been obtained if there had been time to make more than one analysis to obtain an average. The need for such an average is evident in the variations in percentage trends. There is enough

Despite the many variables and limitations in an investigation of this sort, quite a few rather definite conclusions may be drawn from it. As an investigation of the practical problem of producing a pure B-elay for further use, it has been shown that such a conversion from an unknown elay to an B-elay can be effected. The definite return of the B-elay to the outside to a known value has to be determined alone indicates rather definitely that some exchange has been carried to completion. Definite proof of this theory was obtained by the spectrographic analysis made by R. V. Brown in his work on spectrographic analysis methods of identifying elays on the sample used in 1904. The disappearance of the solution line indicated that solution had been removed from the elay. The appearance again after separation was probably due to a mixture in procedure. The final sample of elay was taken from the cell the day after the run was completed. This showed the solution and magnesium in the elay water to have through the process of elaying and later the elay mix. By this process the percentage of solution and magnesium increased. Some accurate results could have been obtained if there had been time to make more than one weighing to obtain an average. The need for such an average is evident in the variations in percentage found. There is enough

consistency, however, to allow confidence to be placed in the analysis. It is also felt that the calcium was not completely removed as early as the analysis indicates.

The production of an H-bentonite still remains as a problem. It is felt that such a clay can not be produced in sufficient quantities by the procedures used in this investigation. The thixotropic properties of the clay, along with the exceptionally high current, caused most of the difficulties. This high current is due to the rapid breaking apart of clay lattices. More investigation might reveal that a lower applied voltage would decrease this breakdown and permit a conversion. The limited quantity that can be treated would still be a major drawback.

From the experiments performed it may be stated that there is a definite indication of base exchange completion, the return of the catholyte pH to a normal value. This return should have been expected upon examination of the factors affecting the pH. The only thing affecting the pH, aside from electrolysis, is the adsorbed ions attracted to the cathode alone. The presence of a base, and the adsorbed ions are bases, increases the hydroxal ion concentration causing a higher than normal pH. When these adsorbed ions are no longer removed, the hydroxal ion concentration decreases to that value caused by electrolysis. This furnished the needed end point in the process.

It may also be concluded that the difficulty of electrophoresis can be effectively solved, at least with some clays. By breaking the clay particles down to a small enough size this difficulty is overcome. Such a process is another good example of the validity of the inverse square law. This treatment, as effected by an ultrasonic vibrator, does introduce the limitation of a reduced capacity for the cell. By decreasing the amount of clay that may be held in solution, the dispersion decreases the amount of clay that may be effectively treated at one time. This problem might readily be solved by a cell of larger proportions.

In any further work that might be done on electro-dialysis, a cell of different proportions and make-up should be used. It is the opinion of the author that effective electrode and membrane areas are of primary importance. For this reason a cell of increased relative width and depth and decreased relative length should be used. A more effective cathode membrane should also be sought in order to overcome the embrittlement problem. If this problem could be eliminated one more stumbling block to the treatment of bentonites would be removed.

As a study of the base exchange properties this investigation revealed as many possibilities as conclusions. The necessity of using tap water eliminated the possibility of determining the order of removal by qualita-

It may also be considered that the difficulty of
 electrochemical cells is effectively solved, at least with
 some alloys. By dissolving the alloy particles down to a
 small amount also this difficulty is overcome. Such a
 process is another good example of the utility of the
 inverse square law. This treatment, as effected by an
 alternative vibrator, does introduce the limitation of
 a reduced capacity for the cell. By dissolving the
 amount of alloy that may be added in solution, the dis-
 solution decreases the amount of alloy that may be effec-
 tively treated at one time. This problem might readily
 be solved by a cell of larger proportions.

In any further work that might be done on electro-
 chemical cells, a cell of different proportions and make-up
 should be used. It is the opinion of the author that
 effective electrode and separator areas are of primary
 importance. For this reason a cell of increased ratio
 of electrode area to separator area and decreased relative length should
 be used. A more attractive and more efficient design
 is needed in order to overcome the limitations of the
 present. If this problem could be eliminated some more satis-
 factory use of the system of electrodes would be re-
 solved.

As a study of the basic principles of electro-
 chemical cells is made, the possibilities are numerous.
 The necessity of using the water alkaline cell is
 likely of determining the order of removal of gas.

tive chemical means. Only a sufficient supply of distilled water could eliminate this problem.

A study of the diffraction patterns, however, did seem to substantiate a generally accepted theory concerning ionic adsorption. The straining of the clay lattice by adsorbed hydrogen ions can most logically be explained by the presence of some substance where no substance was before. If there were a simple surface exchange of a hydrogen ion for a calcium ion, this condition would not exist. But the formation of a water molecule by the addition of a hydrogen ion to a hydroxal ion would satisfy the requirement. This belief is further substantiated by the fact that a hydrogen ion heads the lyotropic series for adsorption and is the most difficult to remove.

By analyzing the pH curves against those produced by electrolysis another conclusion may be drawn. The rate of exchange is most rapid at the beginning and continues to decrease up to the end point. This points to the possibility that rate of exchange is more dependent upon the concentration of the adsorbed ion than the substituted ion. This must be concluded because the concentration of the substituted ion remains substantially constant.

One of the most interesting aspects of the investigation was the possibility of studying the order of

exchange by spectrographic analysis. Such a study would not only permit a determination of the order of exchange but also of the comparative rates of exchange. The latter determination might furnish an interesting tie-in with the order of the lyotropic series. Such a study was hampered by the presence of only one adsorbed ion on the clay analyzed.

It is the opinion of the author that considerably more work needs to be done on electrodialysis before a completely satisfactory method of producing a pure H-clay can be obtained. A study of a continuous flow process might be a possible answer. In any event I feel that much more could be accomplished by more than one person working together.

exchange of scientific materials. Such a study would not only serve a definition of the order of research but also of the comparative value of research. The fact that scientific study is not a simple matter of order of the research series, and a study was suggested by the statement of only one example for on the only subject.

It is the opinion of the author that comparatively more work needs to be done on scientific study before a satisfactory method of choosing a new E-ray can be devised. A study of a continuous line process might be a valuable study. In any event I feel that such work could be accomplished by some form of action arising together.

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APPENDIX

INFORMATION FOR RUN #1

Anode - Carbon

Cathode - Monel

Stainless steel stirrer

One cooling grid used

Sample analysis: 200 grams of unknown clay

Sieve #	o/o Passing
200	1.05
140	11.05
60	100.00

Water used:

Center compartment - 925 cc of distilled water

End compartments - 1000 cc of distilled water with
no circulation

DATA FOR RUN #1

TIME (min)	TEMP. °C	VOLT. (v)	CURRENT (a)	pH		AL	CA	MG	NA	K
				CATHODE	ANODE					
0	19	115	0.10	6.55	6.55					
60	19	114	0.30	10.55	5.55					
240	16	113	0.20	11.80	4.20					
570	16	114	0.20	12.00	4.38					
1260	16	111	0.20	12.01	5.40					
1440	16	112	0.25	11.89	4.42					
1920	17	111	0.25	12.19	4.47					
1935		STOPPED				T _c	P _{T_c}	T _c	-	-

"T" denotes trace
 "P" denotes definite
 presence
 Subscript denotes
 electrode.

REMARKS:

Green precipitate identified as the hydroxides of
 nickel and copper formed at the anode.

Clay had a tendency to pack up on the anode membrane.

INFORMATION FOR RUN #2a

Anode - Carbon

Cathode - Monel

Stainless steel stirrer

No cooling grids used

Sample analysis: 200 grams of unknown clay passing a
#35 sieve.

Water used:

Center compartment - 1925 cc of distilled water

End compartments - 2000 cc of tap water with con-
stant circulation.

DATA FOR RUN #2a

TIME (min)	TEMP. °C	VOLTAGE (v)	CURRENT (a)	pH	
				CATHODE	ANODE
0	17	117	0.25		
5	17	117	0.30	10.62	8.78
10	18	117	0.40	10.38	9.00
30	19	114	0.55	10.72	8.82
240	18	110	0.85	10.66	7.60
480	16.5	110	0.70	10.65	7.71

REMARKS:

Overflow pipes clogged by hydroxides of copper and nickel.

End compartments overflowed into center compartment. Inspection of anode showed that half of it had been corroded away.

Inspection of metal stirrer showed that propeller had been corroded away and the rod was very pitted.

DATE	TIME	TEMP. (°C)	VOLTAJE (V)	CURRENT (A)	ATMOSPHERIC PRESSURE (mm Hg)
10.10.70	10.10	10.1	11.1	0.11	10.1
10.10.70	10.20	10.2	11.2	0.12	10.2
10.10.70	10.30	10.3	11.3	0.13	10.3
10.10.70	10.40	10.4	11.4	0.14	10.4
10.10.70	10.50	10.5	11.5	0.15	10.5
10.10.70	11.00	10.6	11.6	0.16	10.6

continued

Overline pipes changed by hydraulic of copper and

alloy.

Red concentration over/under into center compartment.

Concentration of water showed that half of it had been

removed.

Concentration of water showed that half of it had been

removed.

alloy.

INFORMATION FOR RUN #2b

Anode - Carbon

Cathode - Carbon

Glass rod stirrer

No cooling grids used

Sample analysis: 200 grams of unknown clay passing a
#140 sieve.

Water used:

Center compartment - 1925 cc of distilled water

End compartments - 2000 cc of tap water with constant circulation.

1. General - General

2. General - General

3. General - General

4. General - General

5. General - General

6. General - General

7. General - General

8. General - General

9. General - General

10. General - General

DATA FOR RUN #2b

TIME (min)	VOLT. (v)	CURRENT (a)	TEMP. °C	pH	
				CATHODE	ANODE
0	117	0.25	18	8.05	8.59
				8.70	7.93
1	118	0.25	18	8.88	7.90
				8.96	7.82
5	119	0.30	17	9.01	7.83
				9.30	7.72
10	117	0.30	16	9.47	7.63
				9.57	7.79
30	115	0.30	16	9.80	7.57
				9.76	7.47
90	112	0.35	16	10.01	6.05
				10.07	5.86
450	110	1.45	26	10.07	5.67
				10.40	4.24
790	111	1.25	18	10.48	4.09
				10.50	4.09
1280	111	0.90	17	10.98	3.70
				11.03	3.60
2160	109	1.20	17.5	11.00	3.58
				10.71	3.67
2820	110	1.15	18	10.84	3.64
				10.84	3.65
3630	110	1.00	20	10.76	3.70
				10.87	3.66
4200	109	1.20	15	10.91	3.64
				10.86	3.66
				10.98	3.54
				10.93	3.50
				10.66	3.67
				10.79	3.62
				10.71	3.60
				10.63	4.00
				10.73	3.86
				10.69	3.84
				9.80	3.43
				9.88	3.40
				9.88	3.38

SPECTROGRAPHIC ANALYSIS OF SAMPLE #2b

	SiO ₂ %	Al ₂ O ₃ %	Fe ₂ O ₃ %	CaO %	TiO ₂ %	MgO %
Before	57.10	23.40	7.70	1.08	0.78	2.22
1	55.80	23.40	10.05	-	0.80	2.12
5	57.30	24.00	8.01	1.83	0.76	2.12
10	50.15	22.60	15.05	0.96	0.76	2.15
30	54.60	21.30	12.00	0.58	0.79	2.08
150	48.10	24.00	13.41	1.53	0.79	2.54
450	54.40	22.30	9.80	1.61	0.82	2.23
790	56.00	23.50	8.40	-	0.80	2.13
1280	55.90	24.00	14.50	-	0.76	2.01
2160	59.00	25.40	8.85	-	0.82	1.56
After	56.50	25.40	8.48	0.71	0.74	2.03

There was no sodium or potassium present. The remainder of the sample was lost on ignition.

ANALYSIS OF SAMPLE 479

SiO ₂ %	Al ₂ O ₃ %	Fe ₂ O ₃ %	CaO %	TiO ₂ %	MgO %
67.10	13.40	7.70	1.08	0.70	1.12
66.35	13.40	10.00	-	0.80	0.12
65.30	14.00	9.01	1.82	0.70	0.12
65.12	13.80	13.02	0.96	0.70	0.12
64.40	11.50	12.00	0.98	0.70	0.09
64.10	12.00	12.41	1.22	0.70	0.04
64.40	12.00	9.00	1.81	0.68	0.12
64.00	12.80	8.42	-	0.80	0.12
63.40	12.00	14.00	-	0.70	0.01
63.00	12.40	8.80	-	0.82	0.04
62.50	12.40	8.48	0.71	0.74	0.02

There was no sodium or potassium present. The
remainder of the sample was lost in digestion.

INFORMATION FOR RUN #3

Anode - Carbon

Cathode - Carbon

Glass rod stirrer

No cooling grids used

Sample analysis: 200 grams of kaolin passing a #60 sieve

Water used:

Center compartment - 1925 cc of distilled water

End compartments - 2000 cc of tap water with constant circulation.

DATA FOR RUN #3

TIME (min)	TEMP. °C	VOLT. (v)	CURRENT (a)	pH	
				CATHODE	ANODE
0	20			7.00	6.78
				7.10	6.71
1	20	113		7.00	6.96
				7.25	6.80
				8.22	7.30
3	19	113	0.10	8.57	7.10
				8.52	6.96
				8.40	7.30
6	19	113	0.10	8.61	7.12
				8.82	6.96
				8.94	7.78
10	19	113	0.15	9.02	7.50
				9.11	7.26
				9.03	7.52
20	18	112	0.15	9.28	7.20
				9.36	6.92
				9.16	7.02
55	17	113	0.15	9.40	6.58
				9.52	6.13
				6.92	3.81
415	16	114	0.25	7.09	3.93
				7.35	4.10
				8.43	7.52
960	13.5	108	0.15	8.63	7.39
				8.73	7.27
				9.52	7.94
1460	15	111	0.20	9.63	7.09
				9.70	6.96
				9.33	7.38
1820	15	109	0.20	9.43	6.94
				9.53	6.60
				9.41	6.22
2365	15	108	0.20	9.49	5.92
				9.56	5.60
				10.22	3.38
3310	19	108	0.30	10.22	3.28
				10.28	3.23
				10.22	3.62
3855	17	110	0.30	10.24	3.21
				10.29	3.94
				9.99	4.13
4185	17	113	0.20	10.02	4.02
				10.04	4.00

REMARKS:

Minimum readings at time 415 due to failure of circulating water supply at that time.

DIFFRACTION DATA FOR RUN #3

Before Treatment

Ring #	Radii (mm)		Diameter (mm)
1	1.21	1.22	2.43
2	1.73	1.73	3.43
3	1.90	1.91	3.81
4	2.25	2.26	4.51
5	2.73	2.73	5.46
6	3.09	3.09	6.18
7-BR	5.95	6.00	11.95

After Treatment

1	1.30	1.30	2.60
2	1.83	1.83	3.66
3	1.99	2.00	3.99
4	2.36	2.38	4.74
5	2.84	2.86	5.70
6	3.20	3.22	6.42
7-BR	5.99	6.08	12.07

BR denotes a back reflection.

DISPERSED DATA FOR RUN 43

Before Treatment

Class	Depth (mm)	Dispersed (cm)
1	1.15	1.15
2	1.75	1.75
3	1.90	1.91
4	2.25	2.10
5	2.75	2.45
6	3.75	3.15
7-10	4.75	11.90

After Treatment

1	1.15	1.15
2	1.75	1.75
3	1.90	1.90
4	2.25	2.15
5	2.75	2.75
6	3.75	3.15
7-10	4.75	12.05

IN duplicate a back reflection

INFORMATION FOR RUN #4

Anode - Carbon

Cathode - Carbon

Glass rod stirrer

No cooling grids used for the first nine minutes and then two were added.

Sample analysis: 200 grams of Osage, Wyoming, bentonite, passing a #60 sieve for the first nine minutes, after which 28 grams were removed.

Water used:

Center compartment - 1925 cc of distilled water for the first nine minutes after which 10 more cc were added.

End compartments - 2000 cc of tap water with constant circulation.

Index - 10000

Index - 10000

Index - 10000

Index - 10000

Index - 10000

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Index - 10000

DATA FOR RUN #4

TIME (min)	TEMP. °C	VOLT. (v)	CURRENT (a)	pH	
				CATHODE	ANODE
0	18	108	6.7		
1	18	105	8.0	13	0
5			10.0		
9		STOPPED			

REMARKS:

- 1 minute - clay proved very thixotropic. Much gas liberated at anode and cathode.
- 5 minutes - clay mix liquid at cathode membrane but not at anode membrane.
- 7 minutes - much steam being liberated from clay mix.
- 9 minutes - clay mix too hot, 75 °C.

Removed 450 cc of clay mix and replaced with 450 cc of distilled water. Cooling grids added.

TIME (min)	TEMP. °C	VOLT. (v)	CURRENT (a)	pH	
				CATHODE	ANODE
0	18	108	6.4	9.33	8.70
				9.10	8.47
1	18	108	6.6	11.25	3.30
				11.49	3.22
5	18	105	6.8	11.75	3.12
				11.75	3.12
10	18.5	106	7.0	11.76	3.18
				11.74	3.12
30	20	107	5.0	11.92	3.10
				11.90	3.02
110	26.5	110	4.0	11.42	3.69
				11.30	3.40
425	25	109	4.0		3.48
					3.43

REMARKS:

- 1 minute - clay mix liquid at cathode membrane.
- 5 minutes - much steam being liberated from clay mix. clay mix at 60 °C.
- 10 minutes - much gas liberated from anode and cathode. clay mix at 80 °C.
- 30 minutes - clay mix at 70 °C.
- 110 minutes - clay mix at 50 °C.
- 425 minutes - leak developed in cathode membrane.

INFORMATION FOR RUN #5

Anode - Carbon

Cathode - Carbon

Glass rod stirrer

No cooling grids used

Sample analysis: 200 grams of unknown clay passing a #60 sieve, vibrated in solution for 1 minute at 1000 kilocycles per second.

Water used:

Center compartment - 1925 cc of distilled water.

End compartments - 2000 cc of tap water with constant circulation.

DATA FOR RUN #5

TIME (min)	TEMP. °C	VOLT. (v)	CURRENT (a)	pH		CLAY MIX
				CATHODE	ANODE	
0	19	116	0.30	7.00	7.06	
				7.02	7.06	
1	19	116	0.30	8.00	8.06	
				9.00	7.53	
5	19	116	0.30	10.04	7.95	
				10.16	6.48	
10	19	116	0.30	10.42	6.46	
				10.50	4.45	
30	17	115	0.35	10.66	4.02	
				10.63	3.78	
60	17	113	0.30	10.69	3.71	
				10.66	3.70	
265	18	111	0.55	11.28	3.39	
				11.32	3.39	
430	18	113	0.60	11.30	3.33	
				11.27	3.23	
Power supply off approximately 1 hour.						
945	16	112	0.30	10.12	4.53	
				10.19	4.38	
1520	19	111	0.60	11.07	3.30	
				11.14	3.24	
1815	15	110	0.40	10.70	3.30	7.26
				10.71	3.70	7.45
Power supply off approximately 1 hour.						
2345	15	112	0.45	10.75	3.98	7.37
				10.80	3.81	7.53
2540	18	111	0.70	11.26	3.22	7.06
				11.26	3.16	7.18
3215	18	108	0.50	10.90	3.47	6.52
				11.00	3.40	6.70
3695	23.5	109	0.50	11.23	2.77	6.70
				11.12	3.09	6.78
4580	22	111	0.35	10.52	3.72	3.58
				10.48	3.63	3.48
5175	17	108	0.30	10.23	3.44	4.03
				10.34	3.41	3.93

REMARKS:

Much gas was liberated at cathode and a small amount at anode. There was no clay packed on the membranes at any time. Clay appeared at cathode at time 4580 signifying a leak.

DIFFRACTION DATA FOR RUN #5

After Vibration, Before Treatment

Ring #	Radii (mm)		Diameter (mm)
1	1.36	1.35	2.71
2	2.05	2.04	4.09
3	2.58	2.59	5.17
4	3.07	3.07	6.14
5	3.46	3.47	6.93
6-BR	4.81	4.79	9.60
7-BR	5.75		11.50
8-BR	7.68		15.36

After Treatment, 4580 Minutes

1	1.39	1.40	2.79
2	2.06	2.07	4.13
3	2.58	2.59	5.17
4	3.09	3.10	6.19
5	3.49	3.50	6.99
6-BR	4.82	4.84	9.66

After Treatment, 5175 Minutes

1	1.40	1.40	2.80
2	2.05	2.05	4.10
3	2.60	2.60	5.20
4	3.07	3.08	6.15
5	3.48	3.49	6.97

Station Vibration, Torsion Transducer

Station #	Amplitude (mm)	Frequency (Hz)
1	1.25	1.25
2	1.75	1.75
3	2.25	2.25
4	2.75	2.75
5	3.25	3.25
6	3.75	3.75
7	4.25	4.25
8	4.75	4.75
9	5.25	5.25
10	5.75	5.75

Station Vibration, Torsion Transducer

1	1.25	1.25
2	1.75	1.75
3	2.25	2.25
4	2.75	2.75
5	3.25	3.25
6	3.75	3.75
7	4.25	4.25

Station Vibration, Torsion Transducer

1	1.25	1.25
2	1.75	1.75
3	2.25	2.25
4	2.75	2.75
5	3.25	3.25

DATA (Cont'd.)

Ring #	Radii (mm)		Diameter (mm)
6-BR	4.83	4.84	9.67
7-BR	5.78		11.56
8-BR	7.68		15.36

BR denotes a back reflection.

DATA (Cont'd)

Run #	Radius (mm)	Diameter (mm)
6-28	4.45	8.90
7-28	5.75	11.50
8-28	7.85	15.70

SS denotes a good reflection.

INFORMATION FOR RUN #6

Anode - Carbon

Cathode - Carbon

Glass rod stirrer

Sample analysis: no clay was used in this run.

Water used:

Center compartment - 2000 cc of distilled water

End compartments - 2000 cc of tap water with constant circulation.

DATA FOR RUN #6

TIME (min)	TEMP. °C	VOLT. (v)	CURRENT (a)	pH	
				CATHODE	ANODE
0	19.5	115	0.00	7.03	5.13
				7.22	4.90
1	19.5	115	0.00	6.72	6.20
				6.82	6.05
5	17	113	0.05	6.80	6.37
				6.85	6.20
10	17	113	0.05	6.60	6.52
				6.82	6.47
30	17	114	0.05	6.92	6.91
				7.07	6.62
110	17.5	114	0.10	6.18	7.00
				8.61	6.42
240	17.5	110	0.25	9.65	4.72
				9.82	4.30
650	18	107	0.35	10.20	5.44
				10.12	5.40
1170	17	107	0.25	10.15	3.93
				10.23	3.76
1380	16.5	107	0.30	10.42	3.82
				10.46	3.56

Power supply off approximately 1½ hours.

1790	16.5	112	0.25	8.82	6.02
				9.58	5.60
1965	16.5	109	0.25	9.78	4.35
				9.86	4.30
2490	16	107	0.30	10.04	3.33
				10.05	3.33
3055	17	103	0.30	10.32	3.93
				10.32	3.38
3400	17	107	0.30	10.10	3.36
				10.04	3.82
3960	16	103	0.30	10.40	3.92
				10.32	3.82
4170	17	106	0.50	10.50	3.52
				10.60	3.45

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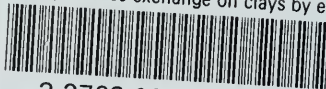
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